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Steady State Calorimetric Measurement of Total Hemispherical Emittance of Cylindrical Absorber Samples at Operating Temperature

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Abstract. At DLR's QUARZ Center a test bench has been established to measure, using steady state calorimetric method, the total hemispherical emittance of cylindrical solar thermal absorber samples at temperatures up to 450 °C. Emittance measurement of solar absorber surfaces is commonly performed by direct-hemispherical reflectance measurements with spectrophotometers. However, the measurement of cylindrical samples with spectrophotometers can be considered still a challenge as integrating spheres, reference samples and calibration services by national metrology institutions are optimized for flat sample measurement. Additionally samples are typically measured at room temperature. The steady state calorimetric method does not rely on reference samples and the measurement is performed at operating temperature. In the steady state calorimetric method electrical power input used to heat the sample is equated to the radiative heat loss from a heated sample to the environment. The total emittance can be calculated using the Stefan-Boltzmann equation from radiative heat loss power, the defined sample surface area and measured surface temperature. The expanded uncertainty ($k=2$) of the total hemispherical emittance has been determined to $\pm 13\%$ for a typical parabolic trough absorber sample at a temperature of 300 °C and a heating power of 100 W. The test bench was validated by the measurement of three samples with the spectrophotometer and the steady state calorimetric method.

INTRODUCTION

A key component of a parabolic trough plant is the solar receiver. The receiver is an absorber enclosed by a glass envelope. The absorber is a tube of stainless steel with a selective coating. As the annulus between absorber and glass envelope is evacuated, the main process for heat conduction from the absorber to the glass envelope is radiation and the heat loss power of a modern solar receiver primarily depends on the emittance of the absorber coating and absorber temperature [1], [2] underlining the importance of absorber emittance measurements.

The measurement of emittance of solar absorber surfaces is commonly performed by measurements of direct-hemispherical reflectance, also sometimes hemispherical-direct reflectance [2], with spectrophotometers at room temperature using conservation of energy, Kirchhoff's law of thermal radiation, and weighting with the blackbody spectrum at the target temperature [2], [3]. This is a fast technique, however, there are disadvantages: The angular dependence of integrating sphere efficiency [4] can distort the measurement and can make the measurement sensitive for sample alignment. Often the measurement is performed without a sample heating [3] and the temperature dependence of coating properties or high temperature stability of the coating are neglected, although this can be addressed by heated sample holders. Furthermore national metrological institutions measure flat samples. The transfer to curved references and hence the measurement of curved samples is associated with additional uncertainties.

Steady state calorimetry is another method commonly used in measurements of the total hemispherical emittance of coatings on substrates, metals other solid materials [5], [6]. As the steady state calorimetry does not rely on external reference samples, is not sensitive to angular dependence of reflectance and the heating of the sample is an integral part of the measurement, a test bench for the application in solar thermal was realized at DLR's QUARZ Center, Cologne, Germany. The test bench in its current setup is designed for absorber samples with 70 mm outside diameter and 500 mm length at operating temperatures as used in parabolic trough receivers.

PRINCIPLE OF MEASUREMENT

The total hemispherical emittance is measured by heating a sample (1) to the temperature of interest, measuring electrical input power $P_{electric}$, and equating the radiative heat loss from the heated sample (1) to the environment (2). Hence, electrical heaters are placed inside the sample and thermocouples are used for the measurement of the temperature of the sample inside surface. In the steady state of desired constant temperature of the sample T_1 and electrical power input $P_{electric}$ is identical to the radiative heat loss power over the surface of the sample P_{1-2} corrected by several small parasitic loss terms:

$$P_{1-2} = P_{electric} - P_{wires} - P_{gap} - P_{gas} . \quad (1)$$

The parasitic loss terms are heat loss through the lead wires P_{wires} for the heaters and the thermocouples, heat flux loss via the small gap at the end of the sample P_{gap} and heat loss through gas P_{gas} .

Figure 1 shows the cylindrical sample (1) surrounded by an artificial sky (2), which creates a defined radiation exchange. Provided that the surface of the artificial sky isn't a black body, multiple reflexions between both surfaces exist.

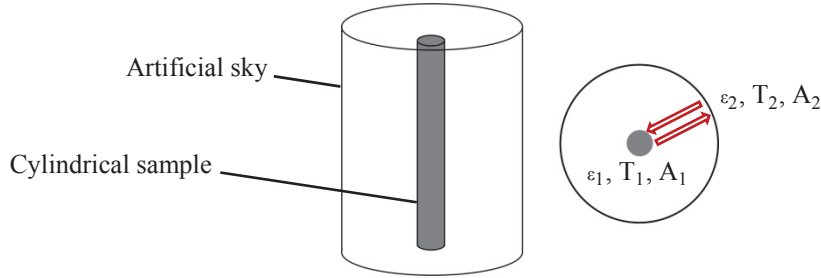


FIGURE 1. Basic geometry of steady state calorimetric method

Emittance ε can be calculated via the Stefan-Boltzmann equation using radiative heat loss power P_{1-2} , sample surface area A_1 and surface temperature T_1 . Approximating the configuration as concentric cylinders of infinite length, grey bodies and diffuse reflection at surface A_2 , the net-heat flux from sample to artificial sky can be calculated [7]. As this test bench is specifically intended to be used for selective coatings the equation has been modified by separate evaluation for both the radiation originating at surface 1 and the radiation originating at surface 2. This allows for using two different emittances at both temperatures with $\varepsilon_{1(T_1)}$ $\varepsilon_{1(T_2)}$ for surface A_1 and $\varepsilon_{2(T_1)}$ $\varepsilon_{2(T_2)}$ for surface A_2 :

$$P_{(1-2)} = A_1 \cdot \sigma \cdot \left[\frac{T_1^4}{\frac{1}{\varepsilon_{1(T_1)}} + \frac{A_1}{A_2} \left(\frac{1}{\varepsilon_{2(T_1)}} - 1 \right)} - \frac{T_2^4}{\frac{1}{\varepsilon_{1(T_2)}} + \frac{A_1}{A_2} \left(\frac{1}{\varepsilon_{2(T_2)}} - 1 \right)} \right] . \quad (2)$$

By solving Equation 2 towards Emittance $\varepsilon_{1(T_1)}$ the total hemispherical emittance of the sample surface can be calculated with the Stefan-Boltzmann constant σ ($\text{kg s}^{-3} \text{K}^{-4}$), the measured surface temperatures T_1 and T_2 (K), the calculated surface areas of A_1 and A_2 (m^2), the known emittances $\varepsilon_{2(T_1)}$ and $\varepsilon_{2(T_2)}$ of surface A_2 and the emittance of $\varepsilon_{1(T_2)}$ calculated by the measurement point with the lowest temperature without a grey body correction.

EXPERIMENTAL SETUP

The experimental setup for measuring the total hemispherical emittance is depicted in Fig. 2 (a). The measurement is performed within a cylindrical vacuum chamber of 0.8 m in diameter and 1.0 m in height. Vacuum pressure is measured and kept at $p < 10^{-4}$ mbar during heat-up and measurement. Heat loss through gas P_{gas} is calculated according to [5] based on the pressure measurements. The inner surface of the artificial sky of 50 cm in diameter and 70 cm in height is coated with Senotherm UHT 600 with an emittance of 0.8 ± 0.1 at room temperature in order to achieve a high total hemispherical absorptance of surface A_2 . The artificial sky is equipped with a water cooling system covering the whole surface.

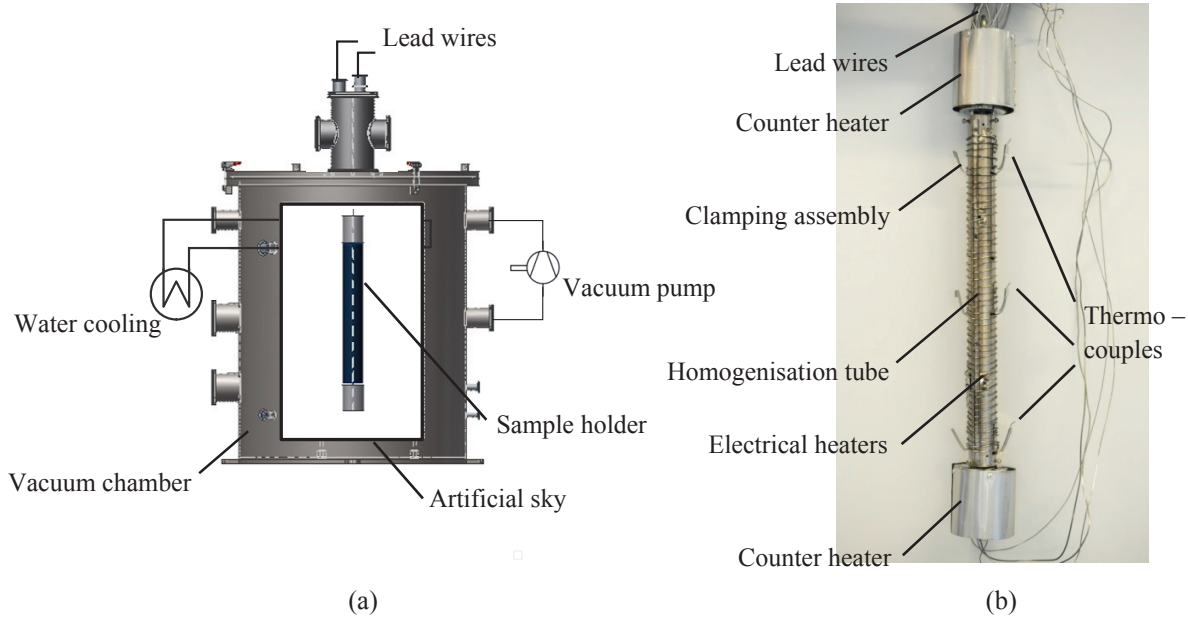


FIGURE 2. (a) Sketch of experimental setup. (b) Sample holder design

Central element of the test bench is the sample holder shown in Fig. 2 (b). The sample holder positions the sample concentrically inside the artificial sky. Where possible the sample holder is made of stainless-steel. The sample holder can support a cylindrical sample of 500 mm in length and inside diameter of 66 mm, the typical dimensions of absorber tubes with 70 mm outside diameter. The sample is held by six clamping assemblies made of spring leaves and is heated by radiation from the electrical heaters and the homogenisation tube. Three electrical heaters are wrapped around the homogenisation tube and can be regulated separately with three heating zones at top, middle and bottom. The powers of the three heaters are measured separately, the sum yields $P_{electric}$. The temperature of the sample is measured with three thermocouples for each heating zone which are pressed at the inner surface of the sample. The axial end faces of the tubular sample are covered with counter heaters to create adiabatic conditions. The counter heaters are clamped on the lead wires of heaters and thermocouples and hence heat the lead wires to minimize heat loss via the lead wires. Heat loss through lead wires P_{wires} is calculated according to [5]:

$$P_{wires} = \pi \cdot \left\{ 0.1 \sigma \cdot \lambda_w \cdot \varepsilon_w \cdot d_w^3 \cdot \left[(T_h^5 - T_{ch}^5) - 5T_2^4 (T_h - T_{ch}) \right] \right\}^{0.5} \quad (3)$$

where λ_w is the thermal conductivity of the lead wire ($\text{W m}^{-1} \text{K}^{-1}$), ε_w is the emittance of the lead wire, d_w is the lead wire diameter (m), T_h is the temperature of the homogenization tube (K) and T_{ch} the temperature of the counter heater inner surface (K).

There is a gap of approx. 5 mm between sample and counter heaters at both ends of the sample, as shown in Fig. 3 (a). To prevent a radiative heat loss of the homogenisation tube, multiple layers of aluminium foil have been mounted in the gap. Nevertheless this radiation protection shields are heated up via inevitable contact to the homogenisation tube and to the counter heaters end surface. To take the radiative heat loss of the radiation protection shields into account, Equation 1 has been supplemented with the heat flux loss value P_{gap} using:

$$P_{gap} = 0.5 \cdot \sigma \cdot A_g \frac{(T_g^4 - T_2^4)}{\frac{1}{\varepsilon_g} + \frac{A_g}{A_2} \left(\frac{1}{\varepsilon_{2(T_2)}} - 1 \right)} \quad (4)$$

where A_g is the surface area of the gap (m^2), T_g is the surface temperature of the gap (K), T_2 is the temperature of the artificial sky (K), ε_g is the emittance of the gap, A_2 is the surface of artificial sky (m^2) and $\varepsilon_{2(T_2)}$ is the emittance of the artificial sky. The dissipation value of P_{gap} is calculated under the assumption that the radiation protection shields are a cavity with the emittance of aluminium foil $\varepsilon_g = 0.5$, the surface temperature of the gap T_g is equal to the sample surface temperature T_1 , the surface area of the gap A_g is approx. 20 cm^2 and the radiative heat loss of the protection shields are caused by one half to the counter heaters and by the other half to the homogenisation tube or rather by the electrical heaters of the sample.

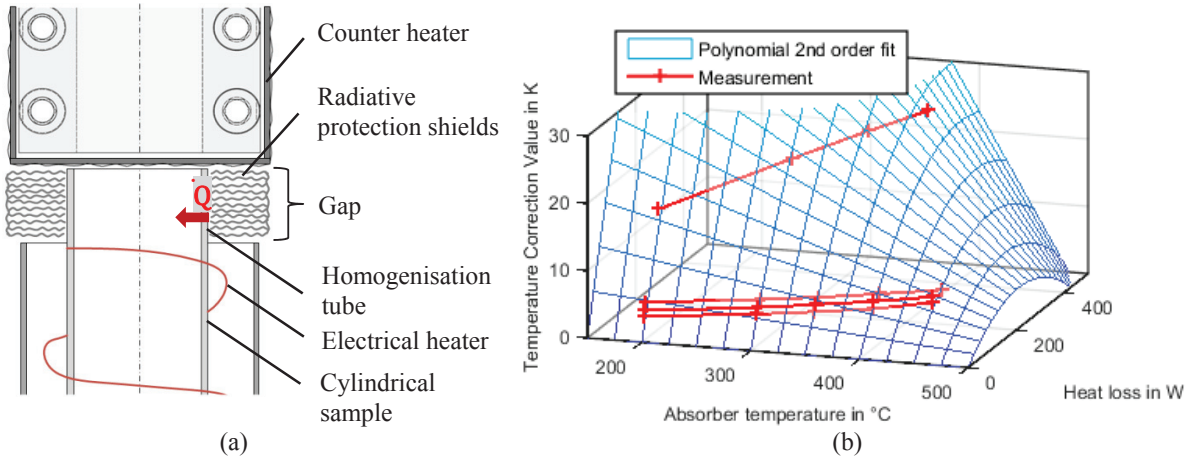


FIGURE 3. (a) Sketch of radiative heat losses of a gap between sample and counter heaters. (b) Plot of Polynomial 2nd Order - Fit of temperature correction

The temperature measured by the thermocouples $T_{1,meas}$ is not used directly as showing the absorber temperature as the thermocouples are influenced by thermal conduction via the lead wires and the radiation temperature in the annulus between sample and homogenisation tube. Hence the thermocouples indicate a temperature that is higher than the temperature of the absorber inner surface. This deviation is characterized with a calibration tube and the result is used to correct the measurement of a sample. The calibration tube is a stainless steel cylindrical tube with three thermocouples soldered on the outer surface and wrapped with insulation. The thermocouples on the calibration tube are soldered opposite to the tube wall at the positions where the thermocouples from the sample holder touch the inner side. Thus for each position of a thermocouple a temperature offset can be measured. The temperature offset dT_{corr} is assumed as a function of the measured temperature $T_{1,meas}$ and of the heat loss P_{1-2} . Different heat loss P_{1-2} at identical temperature can be realised with a variation in isolation, such as one or more layers of aluminium foil wrapped around the calibrator.

In order to provide a general correction function dT_{corr} for use in the measurement of a sample, the measured temperature difference between inner and outer thermocouples are averaged for the tube and a fit to a model is determined. The model used is an empirical 2nd order, 2-dimensional polynomial with an additional assumption that the correction dT_{corr} must be zero, if the heat loss P_{1-2} is zero. A fit of this model to the calibration data is shown in Fig. 3 (b). The best fit yields

$$dT_{corr}(P_{1-2}, T_{1, meas}) = -1.33 \times 10^{-4} K/W^2 \cdot P_{1-2}^2 - 5.54 \times 10^{-4} K/^\circ C W \cdot T_{1, meas} \cdot P_{1-2} + 0.333 K/W \cdot P_{1-2}. \quad (5)$$

With this correction function dT_{corr} , the best estimate for the outer surface temperature of the sample T_1 can be calculated from the measured temperature and absorber surface heat loss P_{1-2} with

$$T_1 = T_{1, meas} - dT_{corr}(P_{1-2}, T_{1, meas}). \quad (6)$$

The uncertainty of the temperature correction function has been calculated to ± 1.4 K. For example as can be seen from Fig. 4 (a), the value of the temperature offset dT_{corr} for a measured temperature $T_{1, meas}$ of about 300 °C and heat loss P_{1-2} of 101 W is 16 K.

Uncertainty of the measurement is determined based on the Guide to the Expression of Uncertainty in Measurement [8]. Table 1 gives a list of a modelled measurement with simplified assumptions illustrating the typical uncertainties. Only the most relevant uncertainty sources are considered and typical values for the measurement of a sample with high emittance (for a parabolic trough receiver) at 300 °C are given.

TABLE 1. Budget of uncertainty with a coverage factor of $k = 2$ for a measurement of a sample temperature of 300 °C and a heating power of 100 W

Parameter		Value, extended uncertainty ($k = 2$)
Surface area of sample	A_1	(0.10996 \pm 0.00004) m ²
Mean inner surface temperature of sample	T_1	(557.8 \pm 3.1) K
General correction function	dT_{corr}	(15.4 \pm 1.4) K
Mean temperature of the artificial sky surface	T_2	(298.2 \pm 1) K
Electrical input power	$P_{electric}$	(100.0 \pm 1.4) W
Surface area of artificial sky	A_2	(1.335 \pm 0.014) m ²
Mean total hemispherical emittance of artificial sky	$\varepsilon_{2, T1}$	0.8 \pm 0.1
Heat loss by conduction of lead wires	P_{wires}	(0.00 \pm 0.02) W
Heat loss by residual gas in chamber	P_{gas}	0.000004 W
Heat flux loss of protection shields in gap	P_{gap}	(4.8 \pm 2.4) W
→Measured emittance	$\varepsilon_{1, T1}$	0.17 \cdot (1 \pm 13 %)

The uncertainties of the surface area of sample A_1 , the surface area of artificial sky A_2 , the mean total hemispherical emittance of artificial sky $\varepsilon_{2, T1}$, the heat loss by conduction of lead wires P_{wires} , the heat loss by residual gas in chamber P_{gas} , the pressure in chamber and also the mean temperature of the artificial sky surface T_2 can be neglected. The biggest source of uncertainty is the temperature measurement of the sample T_1 including the temperature correction dT_{corr} . Also relevant is the uncertainty of P_{gap} that describes the heat flux loss through the gap between the protection shields. The electrical power measurement $P_{electric}$ is associated with a surprisingly high uncertainty for the installed measurement devices showing room for improvement.

VALIDATION

The steady state calorimetric method was validated by measuring absorber samples with the steady state calorimetric method and with the spectrophotometric method in 2016. For this comparison Schott Solar provided three absorber samples with different properties. The spectrophotometric characterizing was performed by Fraunhofer ISE, Germany, courtesy of Andreas Georg and Franz Brucker. No sample heating was used with the spectrophotometric method.

The results are given in Fig. 4. All uncertainties have a coverage factor of $k = 2$ with a level of confidence of 95 %.

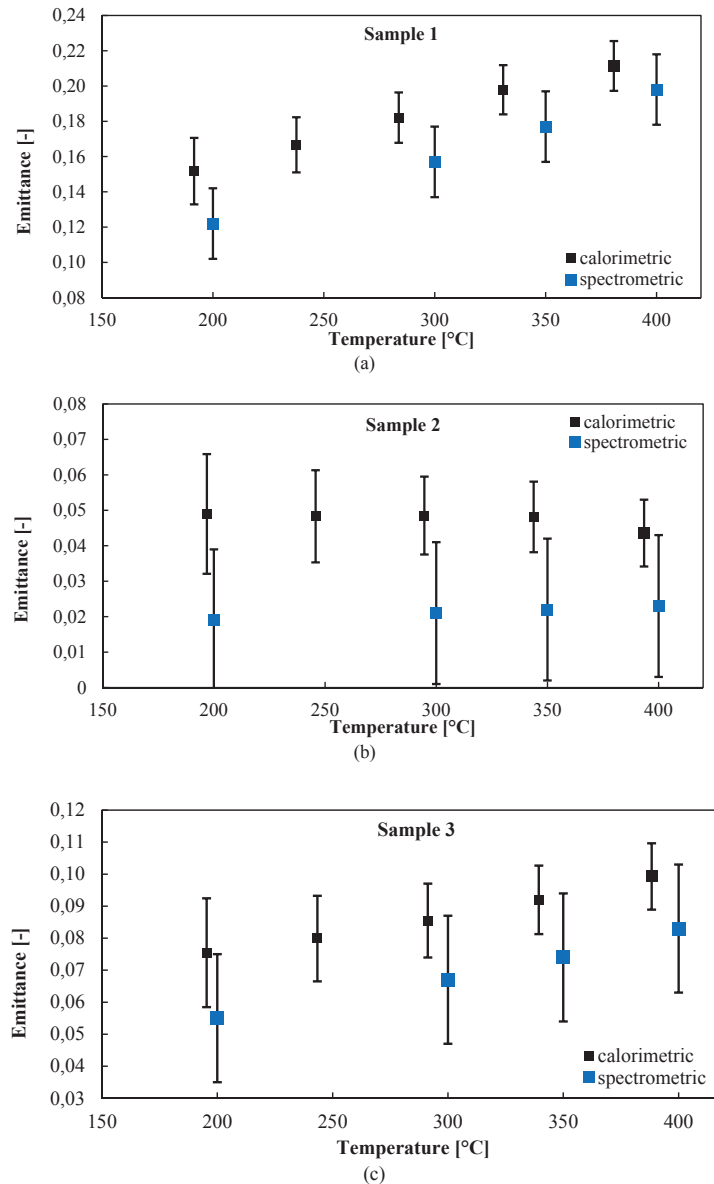


FIGURE 4. Measured emittance with steady state calorimetric method (DLR) and spectrophotometric method (Fraunhofer ISE) for a selectively coated sample of high emittance no.1 (a), a metallic coated sample of low emittance no.2 (b) and a characteristic solar thermal selective absorber sample no.3 (c)

Comparing the test results of sample no.1, the spectrometric method yields an emittance of 0.157 ± 0.02 at 300 °C, while the calorimetric method yields an emittance of 0.186 ± 0.012 at 284 °C. For sample no.2, the spectrometric method yields an emittance of 0.021 ± 0.02 at 300 °C the calorimetric method yields 0.052 ± 0.004 at 295 °C. For sample no.3, the spectrometric method yields an emittance of 0.067 ± 0.02 at 300 °C the calorimetric method yields 0.089 ± 0.008 at 291 °C. Both methods show for samples no.1 und no.3 an increasing emittance with increasing temperature, for sample no.2 no clear trend is visible in the data.

The deviations of the measurements between both methods are within the measurement uncertainties. However, emittances with the steady state calorimetric method are for all samples 0.02 higher than emittances measured with the spectrophotometric method. Discussing only the calorimetric method and assuming than both methods should yield a similar result, this could be caused by incorrect assumptions regarding the calculation of heat flux loss by the gap at the end of the sample P_{gap} , not yet considered heat loss of the homogenisation tube to the vacuum chamber mounting, an inhomogeneous temperature distribution at the sample's outer surface, too high measured electrical power $P_{electric}$ or an overestimation of the temperature measurement error and wrong correction with the calibrator data.

CONCLUSION

DLR's QUARZ Center has developed a new test bench for measuring the total hemispherical emittance of selectively coated solar thermal cylindrical absorber samples. The emittance of tubes of 500 mm in length and inside diameter of 66 mm can be measured at operating temperatures as used in parabolic trough receivers. An uncertainty analysis specifies a measurement uncertainty of the total hemispherical emittance of $\pm 13 \%$ with a coverage factor $k = 2$ for a typical measurement at a sample temperature of 300 °C and a heating power of 100 W.

In a calibration measurement campaign three different absorber samples were measured with the steady state calorimetric method and were characterized by Fraunhofer ISE with the spectrophotometric method without sample heating. All measurements show systematic deviations of about 0.02 to higher measured emittances of the calorimetric method compared to the results of the spectrophotometric method. As all deviations are within the measurement uncertainties, the steady state calorimetric method was successfully validated.

Future work will focus on the interface between counter heater and sample. It is suspected that there is a difference in radial temperature distribution between counter heater and homogenization tube. This effect is currently neglected and needs to be studied further. Also the installed device for measuring the electrical power needs to be improved. Additionally, by reducing the size of the gap between sample and counter heater, heat loss via the gap and associated uncertainties can be reduced. This should lead to a further reduction in overall uncertainty.

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